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## Evaluation of crack initiation criterion at dissimilar interface edge with nanoscale singular stress field

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### Abstract

In the present research, to investigate the crack initiation criterion at the interfacial edge with a nanoscale singular stress field, we conduct mechanical experiments using four kinds of nano-cantilever-shaped specimens where a crack is initiated at the copper/silicon nitride interface. The results reveal that regardless of the specimen dimensions,  $K_{\text{dissimilar interface edge (C)}}$  is constant ( $112 \text{ MPa} \cdot \text{m}^{0.179}$ ) within the nanoscale singular stress field. The interface crack initiation is dominated by the nanoscale singular stress field near the interface edge in the nano-sized multilayered component.

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### 1. Introduction

In a multilayered structure, stress concentrates at the interface under a load due to the deformation mismatch of dissimilar material [Gradin (1982), Zhixue (2008), D.B.Bogy (1968)] and the singular stress field often appears near the interface edge where the interface meets a surface (Free edge effect) [Seiji Ioka et al. (2007), J.H.You and Y.Y.Yang (1998)]. Therefore, the interface edge is one of the potential crack initiation sites. In nanoscale component, once the crack is initiated from the dissimilar interface edge, it immediately leads to malfunction or fracture. Thus, it is important to investigate the criterion for crack initiation from the dissimilar interface edge.

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In a bulk structure, the singular stress field is governed by the stress intensity parameter,  $K_{\text{dissimilar interface edge}}$  near the dissimilar interface edge, and an interfacial crack is initiated at a critical magnitude of  $K_{\text{dissimilar interface edge}}$  from the interface edge [A.R.Akisanaya and C.S.Meng (2003), E.D.Reedy JR (1990), D.Munz and Y.Y.Yang (1993)]. These concepts are based on the continuum mechanics where the singular stress field consists of sufficient number of atoms. As the component size shrinks, the singular stress region near the dissimilar interface edge scales down into the nanoscale [T.L.Becker Jr, et al. (1997), Takashi Sumigawa and Tetsuya Shishido et al. (2010)], which correspond to about several dozen to hundred atoms. In this case, it is not experimentally investigated well whether the interface crack initiation is governed by the nano-sized singular stress field near the interface edge or not.

The purpose of this study is to investigate the crack initiation criterion from the dissimilar interface edge (Cu/SiN) in nanoscale multilayered component on the basis of *in situ* experimental observation.

## 2. Experiment and analysis

### 2.1. Material

The material is a multilayered (Ti/Cu/SiN) thin films formed on a silicon substrate. Titanium (Ti) is deposited up to the thickness of a few nanometers at the rate of 20 nm/min on Si wafer with (100) orientation before a Cu layer is deposited up to the thickness of 200nm at the rate of 25 nm/min by magnetron sputtering. A silicon nitride (SiN) layer is sequentially deposited up to thickness of 900 nm at the rate of 11 nm/min by magnetron sputtering without breaking the vacuum. As the interfacial strength between Si and Cu is increased by depositing Ti thin layer, the interface crack appears at the Cu/SiN interface.

### 2.2. Fabrication procedure

Figure 1 shows the schematic illustration of the fabrication procedure of the nanoscale cantilever-shaped specimens. A  $10\ \mu\text{m} \times 10\ \mu\text{m} \times 10\ \mu\text{m}$  block is cut from the multilayered (Si/Ti/Cu/SiN) material and is picked up by a probe manipulator (Fig. 1(a)). After the block is mounted onto the top of a gold (Au) wire ( $\phi 0.25\ \text{mm}$ ) with a flat top using a tungsten (W) deposition (Fig. 1(b)), the cantilever-shaped specimen containing the Si/Ti/Cu/SiN interfaces is processed by a focused ion beam (FIB: FB-2100FIB system (HITACHI)) (Fig. 1(c)). The gallium (Ga) ion beam energy is set at 40 kV and the beam current is changed from 10 pA to 10 nA depending on the fabrication precision.

Figure 2 shows the schematic illustrations and dimension of the cantilever specimen and the loading scheme for the evaluation of crack initiation criterion at the Cu/SiN interface edge. Four specimens (Specimens 1, 2, 3, and 4) of different sizes, as summarized in Table 1, are prepared.

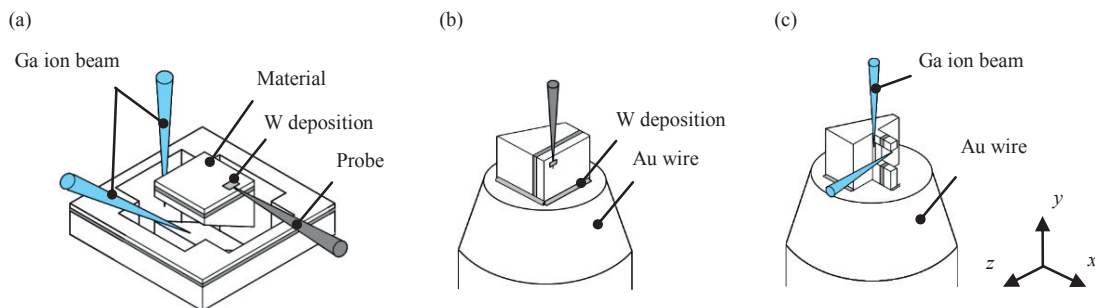


Fig. 1. Schematic illustration of fabrication procedure of the nanoscale cantilever-shaped specimen.

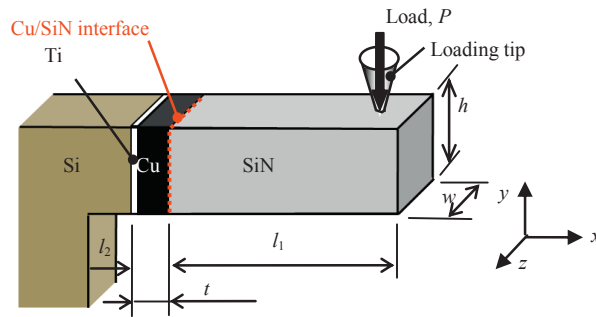


Fig. 2. Schematic illustration and dimension of the nanoscale cantilever-shaped specimen.

Table 1 Multilayered cantilever-shaped specimen size.

Specimen	Height $h$ (nm)	Width $w$ (nm)	Length of SiN $l_1$ (nm)	Length of Si $l_2$ (nm)	Thickness of Cu and Ti thin film $t$ (nm)
1	700	665	897	308	205
2	300	670	888	298	200
3	250	685	896	286	198
4	170	786	929	301	193

### 2.3. Testing system

In order to observe the interface crack behavior, the experiments are conducted in a transmission electron microscopy (TEM; JEOL Ltd. JEM-2100) with accelerating voltage of 200 kV in a vacuum condition of  $2.0 \times 10^{-5}$  Pa. Fig. 3(a) shows the schematic illustration of testing system. The TEM image of the cantilever specimen is continuously recorded through a digital camera (Gatan Inc, ES500) at the frame rate of 60 Hz. The minute mechanical loading apparatus (Nanofactory Instruments AB, SA2000N) which is built into TEM holder used as shown Fig. 3(b). The loading apparatus consists of movable sample stage and a diamond loading tip with a load sensor. The measurement range and accuracy of the loading are 0–1000  $\mu$ N and  $\pm 0.1$   $\mu$ N, respectively. The Au wire, on which a multilayered cantilever-shaped specimen is mounted, is attached to the stage which is actuated three-dimensionally by a piezoelectric actuator. The alignment resolution of the piezoelectric actuator in each direction ( $x$ ,  $y$  and  $z$ ) is approximately 1 nm.

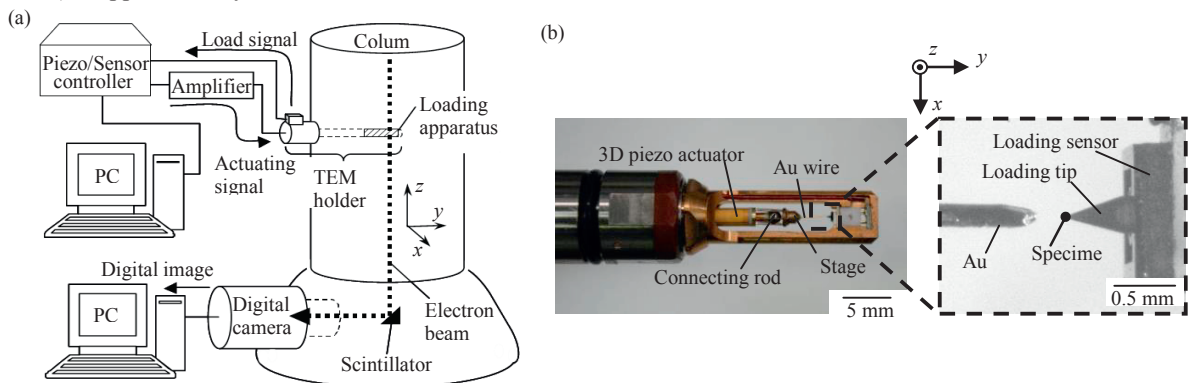


Fig. 3. Schematic illustration of testing system (a) and mechanical loading apparatus built into a TEM holder (b).

### 3. Experimental results

Figure 4 shows the load ( $P$ ) - time ( $t$ ) relationship of Specimen 2 and the TEM images corresponding to points A-C in Fig. 4. The load monotonously increases up to a peak of 39.4  $\mu\text{N}$  (point B) and then suddenly drops near to 0  $\mu\text{N}$  (point C). The magnified TEM image of the Cu/SiN interface edge shows no precursory crack formation at point B. This means that the crack initiates at the point B from the top of Cu/SiN interface edge and instantly propagated along the interface. Similar behavior is observed in other specimens. Therefore, the peak load,  $P_{\text{interfacial crack initiation}}$ , is defined as the crack initiation load. The crack initiation load each specimen is summarized in Table. 2.

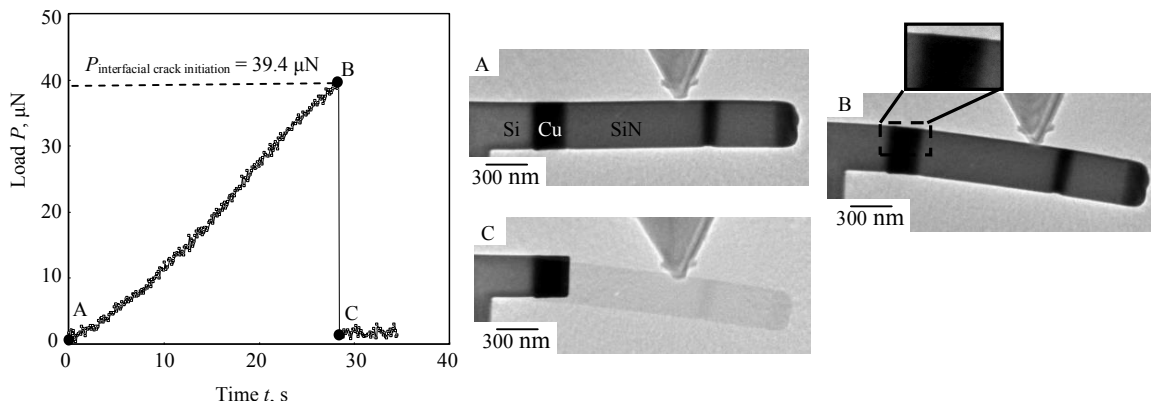


Fig. 4. Loading-time relationship and TEM images corresponding to points A-C.

Table 2 Crack initiation load and bending stress at the interface edge of each specimen.

	Specimen 1	Specimen 2	Specimen 3	Specimen 4
Crack initiation load	173	39.4	21.2	16.2
$P_{\text{crack initiation}} (\mu\text{N})$				

### 4. Crack initiation criterion at Cu/SiN interface edge

#### 4.1. Stress analysis

The stress distribution in the specimen is analyzed by the elasto-plastic finite element method (elasto plastic FEM) analysis, in which an individual cantilever-shaped model is prepared for each experimental specimen reproducing the shape on the base of the 3D analysis of a scanning electron microscope (SEM) and TEM micrographs. Here, Ti layer isn't considered in this analysis model because the thickness is much thinner than that of other layers and it affect little on the stress distribution along the Cu/SiN interface. The region near the dissimilar interfaces (Si/Cu and Cu/SiN), where the stress concentration is expected, is divided into a finer mesh. The perfect constraint condition is imposed on the back and the bottom ends of the model. The Si and SiN are treated as elastic materials because the yield stress of them is specifically high. Tables 3 and 4 show the elastic constants of component materials. The elasto-plastic constitutive equation of the film is given as follows [Takashi Sumigawa and Tadashi Murakami et al. (2010)]:

$$\sigma = \begin{cases} 129000\varepsilon, & \text{for } \sigma \leq 223 \text{ (MPa)} \\ 2106\varepsilon^{0.35}, & \text{for } \sigma \geq 223 \text{ (MPa)} \end{cases} \quad (1)$$

Here,  $\sigma$  and  $\varepsilon$  are the von Mises stress and strain, respectively. In order to examine the effect of plasticity of Cu thin film on the stress distribution near the Cu/SiN interface edge, the elastic FEM analysis is also conducted for Specimen 1.

The residual stresses of Cu and the SiN layers are measured experimentally in a previous paper ( $\sigma_{\text{Cu}} = 147$  MPa,  $\sigma_{\text{SiN}} = -290$  MPa) [Takashi Sumigawa and Tetsuya Shishido et al. (2010)]. The residual stresses are included in the FEM (ABAQUS, ver.6.5-6) calculation because they strongly effect on the stress state near the interfaces.

Table 3 Elastic constants of the component materials (Cu, SiN).

Material	Young's modulus $E$ (GPa)	Poisson's ratio $\nu$
Cu	129	0.34
SiN	197	0.27

Table 4 Elastic constants of the component material (Si).

Material	$C_{11}$ (GPa)	$C_{12}$ (GPa)	$C_{44}$ (GPa)
Si	167.4	65.2	79.6

#### 4.2. Stress distribution along Cu/SiN interface

Figure 5(a) shows the comparison of distribution of normal stress  $\sigma$  along Cu/SiN interface near the edge  $r$  at the crack initiation load,  $P_{\text{interfacial crack initiation}}$  obtained by elasto-plastic FEM analysis and that of the elastic analysis. The difference between the result of elasto-plastic analysis and that of the elastic analysis is appeared. Furthermore, considering the yield stress of Cu thin film is lower than that of Si substrate and SiN layer, the Cu thin film is treated as an elasto-plastic material. Thus, the elasto-plastic analysis is appropriate for calculating the distribution of  $\sigma$  along Cu/SiN interface in this study.

Figure 5(b) shows the distribution of normal stress along the Cu/SiN interface near the edge at the crack initiation load of each specimen obtained by elasto-plastic FEM analysis. This log-log graph shows a linear relationship between  $\sigma$  and  $r$  near the interface edge. Thus, the singular stress field is expressed as follows;

$$\sigma = \frac{K_{\text{dissimilar interface edge}}}{r^\lambda} \quad (2)$$

Here  $K_{\text{dissimilar interface edge}}$  is the stress intensity parameter that characterizes the intensity of the singular stress field near the Cu/SiN interface edge and  $\lambda$  is the order of the stress singularity that depends on the material combination and the edge geometry. It is concluded that regardless of the specimen dimensions, the critical magnitude of the stress intensity parameter,  $K_{\text{dissimilar interface edge (C)}}$  is substantially constant ( $112 \text{ MPa} \cdot \text{m}^{0.179}$ ). Thus, in nano-sized specimens, dissimilar interfacial crack initiation from the interfacial edge with nanoscale singular stress field can be evaluated by criterion of the crack initiation at interface edge based on the concept of the continuum mechanics.

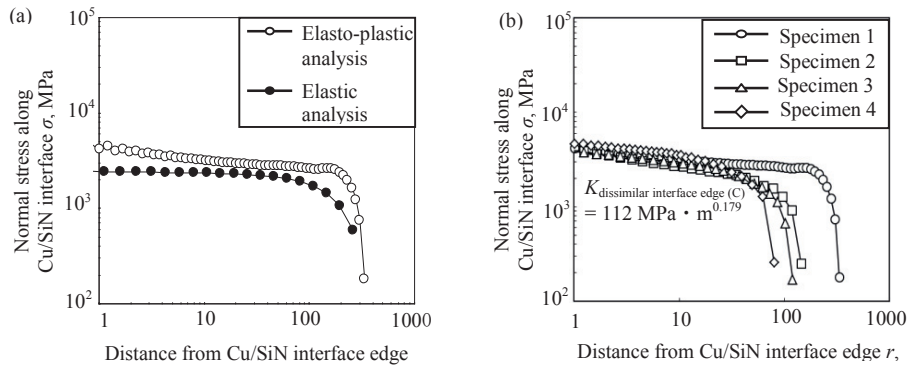


Fig. 5. Distribution of normal stress along the Cu/SiN interface at crack initiation; (a) Comparison the stress distribution obtained by elasto-plastic analysis and that of elastic analysis for Specimen 1 and (b) Obtained by elasto-plastic analysis for each specimen.

## 5. Conclusion

To evaluate the crack initiation from the dissimilar interface edge in nano-sized component with the singular stress field, we conduct mechanical experiments using cantilever-shaped specimens with nano-meter scaled singular stress field at the Cu/SiN interface. The results are summarized as follows:

- (1) At the multilayered cantilevers-shaped specimens (Si/Ti/Cu/SiN) in this paper, a crack is initiated at the Cu/SiN interface edge and the crack instantaneously propagates along the Cu/SiN interface.
- (2)  $K_{\text{dissimilar interface edge (C)}}$  is substantially constant ( $112 \text{ MPa} \cdot \text{m}^{0.179}$ ) within the nano-sized singular stress field regardless of the specimen dimensions.
- (3) The interface crack initiation is govern by the nanoscale singular stress field near the interface edge in the nano-sized multilayered component.

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